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# Range investigation of protons in (100) and (111) silver lattices

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> RANGE INVESTIGATION OF PROTONS IN [100] AND [111] SILVER LATTICES.

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RANGE INVESTIGATION OF PROTONS IN (100) AND (111) SILVER LATTICES

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Terrell N. Cantrell



# RANGE INVESTIGATION OF PROTONS IN (100) AND (111) SILVER LATTICES

by

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Submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN PHYSICS

United States Naval Postgraduate School Monterey, California

1964

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# RANGE INVESTIGATION OF PROTONS IN (100) AND (111) SILVER LATTICES

by

Terrell N. Cantrell

This work is accepted as fulfilling the thesis requirements for the degree of  ${\tt MASTER\ OF\ SCIENCE}$ 

IN

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from the

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#### ABSTRACT

This research utilizes the 2 mev Van de Graaff accelerator as a source of energetic protons to study the range of protons in the (100) and (111) silver lattice structures. The range-energy curve is constructed by plotting target thickness vs the average energy of penetration for several targets of both orientations. This plot reveals there is no detectable difference in the range of protons in Ag with respect to the (100) and (111) crystal orientations in the energy range 200 to 800 KeV.



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### 1. Introduction.

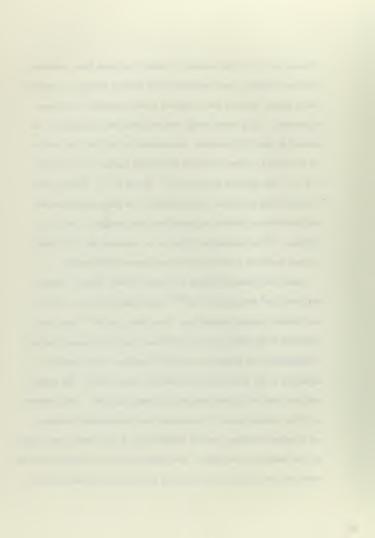
The effect of energetic ions incident on metallic surfaces has been studied by physicists since 1852 when Grove observed the erosion of metallic surfaces under positive ion bombardment in glow discharges. These effects are termed radiation damage. Among the present areas of interest within this field are the study of the average distance of penetration of ions into metals as a function of ion energy, ion mass, target atomic mass, and target lattice structure, and the study of the "sputtering ratio" as a function of these same parameters. The sputtering ratio, or yield, is the number of target atoms ejected per incident ion. The sputtering ratio has also been found to be dependent on the angle of incidence of the ions for both polycrystalline and ordered lattice structure.

Until recently all range studies were made in polycrystalline metals. Range-energy curves for many combinations of ions and polycrystalline targets have been measured, but with the advent of new crystal growing techniques the physicist can now study the effect of lattice structure on ion range. Theoreticians are currently using computers for similar studies. Robinson and Oen<sup>2</sup> using computer calculations showed that with Cu incident of f.c.c. Cu a "channeling" effect took place in the (110) direction.



"Channeling" is the phenomenon of incident particles being deflected into open channels, and consequently the average distance of penetration is greater with the small stopping power associated with these trajectories. For a given energy they predicted the penetration to be greatest in the (110) direction, intermediate in the (100), and smallest in the (111). They also made theoretical studies for Cu incident on b.c.c. and diamond structured Cu. For the b.c.c. structure, the (111) and (100) directions were predicted to be channeling direction with the average distance of penetration being greatest in the (111) direction. In the diamond as in the f.c.c. structure the (110) direction was predicted to be the predominant channeling direction.

Recent experimental studies by Domeij, Brown, Davies, Piercy, and Kornelsen<sup>3</sup> were made for Xe<sup>125</sup> ions incident on b.c.c. W for four different crystal orientations. They found the Xe<sup>125</sup> ions were channeled in the (100) and (111) directions, but the average distance of penetration was greatest in the (100) direction. The range then decreases in the (110) and (112) directions respectively. The experiment was done in an electromagnetic isotope separator. After bombardment thin uniform layers of the crystals were dissolved by anodising and chemical stripping, and the radioactivity of the crystals was measured after removal of each layer. The integral range distributions were obtained by plotting the residual activity against the thickness removed.

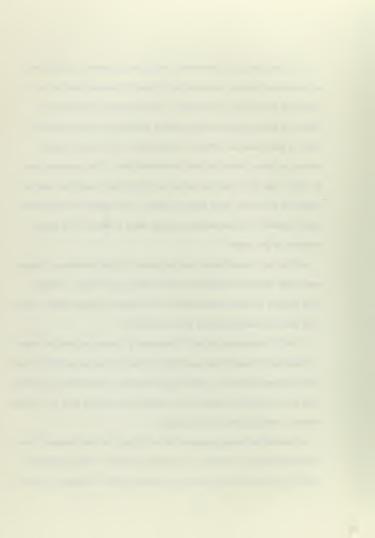


All combinations of bombarding particles and target materials are of theoretical interest, because the physical processes involved in sputtering are not well understood. The differences in behavior between the many possible particle-target combinations yield important clues to the processes involved in sputtering. The energy range of interest is from a few eV up to a few hundred KeV. The sputtering ratio, or yield, rises from very low values at electron-volt energies, reaches a plateau at a few KeV, then drops off agian. The energies at the turning points depend on the bombarding particle mass as well as the lattice structure of the target.

All the early experiments were performed in glow discharges of noble gases with background pressures of the order of  $10^{-3}$  torr. In these dirty systems surface contamination of the target was appreciable, rendering the experimental results quite unreliable.

In 1923 Kingdon and Langmuir <sup>4</sup> proposed a "momentum transfer theory" in attempting to explain the sputtering of thorium from the surface of thoriated tungsten filiments. This theory postulated a depression of a target atom by an incident ion such that a second ion scattered from it can back scatter a thorium atom from the surface.

An alternative theory proposed by von Hippel and Blechschmidt<sup>5</sup> and perfected by Townes<sup>6</sup> was the "evaporation theory", which proposed a local high surface temperature due to the dissipation of energy by the in-



cident ions and the subsequent evaporation of surface atoms.

More recent experiments have been done with ion-beam techniques, using accelerators and mass spectrometers, in which the beam energy and angle of incidence can easily be controlled, secondary electrons can be suppressed, and a high vacuum maintained. Using similar experimental equipment, Wehner has shown that with a given target lattice structure the sputtered atoms are ejected predominatly in the close packed directions. (See Fig. 1).

One goal of physicists in this field is to determine a potential function for a given incident particle and lattice structure, and then be able to predict the effect of energetic ions incident of metallic surfaces. The most common experimental methods of approach today are to study the average distance of penetration of ions into a known lattice structure as a function of energy, to determine the preferred directions of ejected atoms as a function of lattice structure and ion energy, and to determine the sputtering ratio as a function of incident angle and energy for a given lattice structure. To date no potential function has been found to explain satisfactorily the interaction between impinging ion and target lattice. Several potential functions have been proposed, such as, the Born-Mayer, Bohr, and the Thomas-Fermi-Firsov, but none satisfactorily represents the interaction under all circumstances.



As mentioned earlier, experimental data from all possible particle-target combinations are essential for the physicist to understand the physical processes involved in radiation damage. This research was done with protons incident on thin silver films of thickness between 10 thousand and 50 thousand angstroms. The average distance of penetration of protons in the (100) and (111) directions was measured, and the effect of sputtering on the average energy of penetration was studied.



## 2. Experimental Equipment

In all experimental work in radiation damage an ultra high vacuum is highly desirable to prevent surface contamination of the crystal. Therefore, a new all stainless steel system was built to replace the existing system, which had an ultimate vacuum of  $10^{-6}$  torr. The old system consisted of a glass target chamber, a four inch diffusion pump, and a three inch cylindrical, liquid air cold trap between the target chamber and the diffusion pump. This cold trap reduced the pumping speed considerably, thus requiring several hours to reach a pressure of  $10^{-6}$  torr. With the existing system a pressure of  $10^{-7}$  torr is obtained within minutes. This great increase in pumping speed has enabled one to change targets and recycle the system within a few minutes, whereas with the old system, pump-down over night was essential.

The new system consists of a stainless steel target chamber, a six inch diffusion pump, and a six inch, liquid air cooled baffle between the chamber and diffusion pump. (See Fig. 2). The increase in pumping speed can be attributed to the larger diffusion pump and the chevron baffle, which allows a less restricted flow than did the cylindrical trap.

Another highly desirable feature of the new system is, with the exception of quartz view ports and viton o-rings, the all metal construction. This allows the system to be baked out with heating tapes up to a temperature of 400 degrees F., which drives contamination from the inside



surface of the chamber.

In the final construction of the target chamber the top and bottom flanges were welded onto the chamber and machined to mate with the top plate and high vacuum value respectively. These joints were sealed with viton o-rings. The ionization tube port and view ports were also welded onto the chamber. (See Fig. 2). The top plate and target assembly was constructed such that the target had two degrees of translational freedom perpendicular to the beam. (See Fig. 3). The target assembly was connected to the top plate via the conventional o-ring vacuum connectors.

To obtain a lower pressure a cylindrical cold trap 3 1/2 inches in diameter and 9 inches long was placed inside the target chamber. It was made of thin walled stainless steel and reduced to a one inch opening at the top. The trap could not be welded to the half inch thick top plate, since the cold trap wall thickness was very thin. The trap was connected via the conventional o-ring type connector to the top plate, yielding quite satisfactory results. It was found that if the liquid air level in the trap is not too high, or does not stand in the one inch connector tube, then the joint is completely leak tight. This cold trap, when filled with liquid air, lowered the pressure from  $10^{-7}$  to  $5 \times 10^{-8}$  torr. This degassed the system adequately, and thereafter an occasional bake out of 2 to 3 hours was sufficient to maintain the vacuum.



The last and perhaps most critical problem of construction encountered was that of obtaining electrical connections into the vacuum system.' The insulation around the connectors had to offer sufficient electrical resistance to prevent leakage current from passing through the insulator to the top plate. It was found with most connectors that if 1200 volts was applied between the connector and top plate, the resulting current was of the order of  $10^{-6}$  amperes. In the experiment to be discussed the beam current measured was of the order of  $10^{-10}$  to  $10^{-8}$  amperes. Thus for most connectors the leakage current was greater than the beam current by a factor of 103. Finally a "Kovar" glass insulated connector was tested, and its electrical resistance was of the order of 10<sup>14</sup> ohms. This gave a leakage current of  $10^{-11}$  ampers, which was quite reasonable. This electical connector was much larger than most tested, with the glass insulation surrounding the connector being about one inch in outside diameter. It is believed the high electrical resistance of the insulator is due to its larger diameter and not the glass composition, since other smaller glass insulated connectors were tested with no success.

Another very minor problem was encountered, but was easily corrected. The ion gague produced a flux of electrons which struck the target assembly and gave a current of about  $10^{-6}$  amperes. The ion gauge was simply turned off when an experiment was being done. This left the vacuum system with no protection against a leak in the target chamber, but in the



interest of time the situation was not corrected. The problem can easily be solved by placing a negative grid at the mouth of the ion gauge port of potential great enough to drive the electrons back to the ion gauge or chamber wall.

The protection system for the vacuum system is quite elaborate, and is best illustrated by a diagram. (See Fig. 4). The ionization gauge is the primary source of protection. The 500 micron switch offers double protection, and is the sole means of protection if the ionization gauge is not on, as is currently the case during an experiment. If at any time the ionization gauge experiences full scale deflection, the diffusion pump is turned off and the high vacuum valve closed. The foreline valve remains open, and the forepump remains on. If the foreline pressure reaches 500 microns pressure, the diffusion pump is turned off, and the high vacuum and foreline valves are closed. In the case of power failure the same protection is afforded, and in all cases of protection the system remains turned off until the reset mechanism is activated.

The remainder of the experimental equipment consists of the 2 MeV Van de Graaff accelerator and the necessary accompanying equipment. The experimental set up is illustrated by figure 5.



## 3. Experimental Procedure.

Protons from the 2 MeV Van de Graaff accelerator are incident normally upon a thin Ag crystal of known orientation and thickness. The fraction of particles which penetrate the target is a function of energy. This fraction is called N/No, and a plot of this number (after normalization) vs proton energy yields the number-energy curve. N is the number of protons penetrating the target per unit time. This plot gives curves as shown in fugures 7 and 8. It is believed that this curve approximates very closely the integral of a gaussian function. This will be discussed in more detail in the next section. The ratio  $\mathrm{N/N_{\odot}}$  is arrived at by taking the ratio of collector current to one, two, or three times total beam current, depending on whether the beam is mass one  $(H^+)$ , mass two  $(H_2^+)$ , or mass three  $(H_3^+)$ . When these ions strike the target, it is assumed that they break up completely into their constituent protons and electrons. It is further assumed that all primary electrons remain in the target, and that no secondary electrons are ejected from the target or collector. This assumption is good at low energies, but is less correct at higher energies. To correct for this loss of electrons small copper repeller rings were placed in front and behind the target. (See Fig. 3). These rings are placed at a negative 1200 volts potential to repel electrons back to the target.

By definition the energy for which  $N/N_{_{\mbox{\scriptsize O}}}$  is equal to 0.5 is called the average energy of penetration, E $_{_{\mbox{\scriptsize O}}}$ . Likewise the thickness of the crystal



is called the average distance of penetration, or mean range, for protons with this energy. The above experiment is repeated for several crystals of thickness between 10 thousand and 50 thousand angstroms, and for three different positions of each target. This is done for both the (100) and (111) crystal orientations. An average of the average energy of penetration,  $\overline{E}_0$ , is obtained by constructing the numberenergy curve for 3 different positions of each target. The range-energy curve is then constructed by plotting target thickness vs  $\overline{E}_0$  for each target. (See Fig. 9).



## 4. Results.

An example of the number-energy curve is shown in figures 7 and 8. As mentioned earlier, the number-energy curve is thought to approximate the integral of a gaussian function. If this is true, the derivatives computed from the number-energy curve should be normally distributed. The 1604 computer was used to investigate this, and the results are shown in figure 8 in the form of a graph drawn by the computer. The (+)'s represent the experimental slopes computed from the number-energy curve, and the  $(\Delta)$ 's are points of the best fit to the gaussian function. Shown also is the corresponding number-energy curve with a vertical line passing through the average energy of penetration. Therefore, it is concluded that the slope of the number-energy curve is normally distributed, and thus the number-energy curve approximates very closely the integral of a gaussian function.

The results of all the number-energy curves are shown below in tables land 2.

Table 1. (100) Orientation

Target Thickness in Mg/cm <sup>2</sup>	Average Energy of Penetration in kev
1.147 + .016	275 - 4.6
1.373 + .017	317 - 4.3
1.480 + .018	340 + 5.2
1.609 + .019	355 - 2.8
2.760 + .025	550 ± 7.5
5.002 <sup>+</sup> .036	820 -12.5



Table 2. (111) Orientation

Target Thickness in Mg/cm <sup>2</sup>	Average Energy of Penetration in kev
1.319 ± .016	264 + 3.1
1.609 + .018	376 + 8.9
2.224022	453 + 2.2
2.466023	501 + 15.3
3.208 + .027	600 - 17.8

The range-energy curve plotted from the values given in tables 1 and 2 is given in figure 9. The dots represent the (111) data points. The curve was drawn through the (100) points, with the (111) points plotted alongside. A curve through the (111) points would lie almost coincident with the (100) curve. Therefore, the conclusion is that there is no appreciable difference in the mean range of protons in Ag with respect to the (100) and (111) crystal orientations in the energy range 200 to 800 KeV.

A (100) target of thickness  $1.609^{+}$ .  $018 \text{ Mg/cm}^2$  was bombarded at an energy of 367 KeV for 7 hours. A total charge of  $1.67 \times 10^3$  micro-coulombs per cm<sup>2</sup> was incident on the target, corresponding to  $10^{16}$  protons per cm<sup>2</sup>. A subsequent plot of the number-energy curve revealed there was no observable change in the average energy of penetration. A crude experiment was done in the old glass system with a (100) target in which Ag atoms sputtered off the back of the target were collected on a mylar film and irridated with neutrons in the reactor. From the decay curve an estimate was made of the number of Ag atoms



present, and a sputtering ratio of 10 was calculated. If one assumes a sputtering ratio of 10 for the above experiment with the beam current on the order of 10<sup>-10</sup> amperes, a brief calculation shows that about 10 hours are required to sputter away 500 angstroms, which would be barely detectable by a change in the average energy of penetration. The above experiment is certainly not sensitive enough to determine a sputtering ratio, but it is worth noting the excessive time required to erode the surface. This is of considerable practical importance in the field of space travel and ion engine research.

The existing target chamber and vacuum system can now be used with slight modification of the target assembly, to extend this research to investigation of several aspects of the radiation damage problem. Since the (100) crystal has a very smooth surface, a measure of the transmission properties of the different crystal orientations can be obtained by allowing two dimensional rotation of the target. The straggling parameter for different orientations can be measured very accurately by rotating the (100) target in this manner.

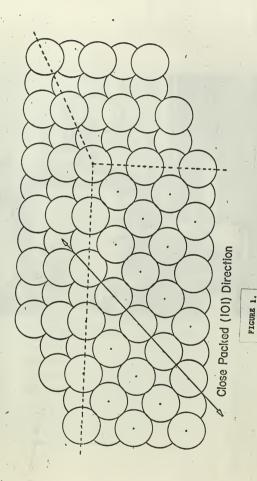
Since Ag can be made radioactive by neutron bombardment, the nuclear reactor available at this school can be used to obtain relative measurements of the sputtering ratio with respect to crystal orientation. This can be done by replacing the Faraday Cup with a collector such as a mylar film to collect the silver atoms sputtered off the back of the



crystal. The collected silver can then be irridated in the reactor, and the decay curve plotted. From this decay an estimate of the number of Ag atoms present can be obtained.

It should be emphasized that I have indicated only a few of the many experiments which can be performed with only slight modification of the target assembly.







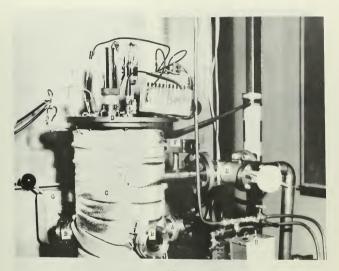


FIGURE 2. STAINLESS STEEL TARGET CHAMBER

A--ION GAUGE PORT

B--VIEW PORTS

C--HEATING TAPE

D--TOP PLATE

E--ROUGHING VALVE

F--VENT VALVE

G--HIGH VACUUM BEAM VALVE

H -- PNEUMATIC HIGH VACUUM VALVE



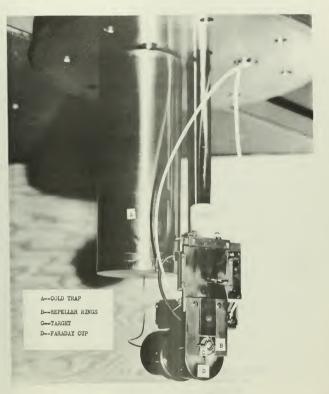


FIGURE 3. TOP PLATE and TARGET ASSEMBLY



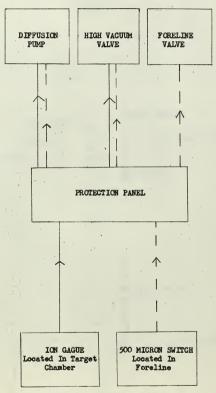


FIGURE 4. Protection system



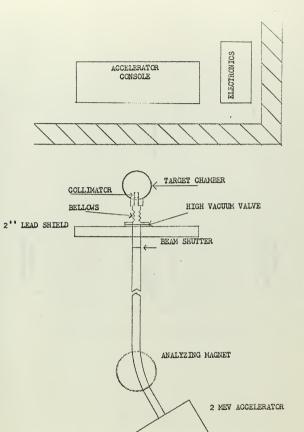
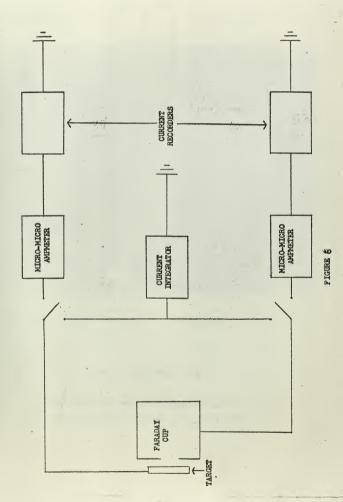


FIGURE 5







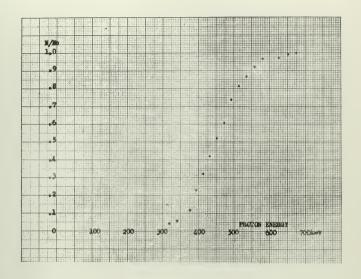


FIGURE 7. NUMBER-ENERGY CURVE

The above graph is an example of the number-energy curve. This curve is for a (111) crystal of thickness 1.103  $^{+}$  0.011 Mg/cm<sup>2</sup>.



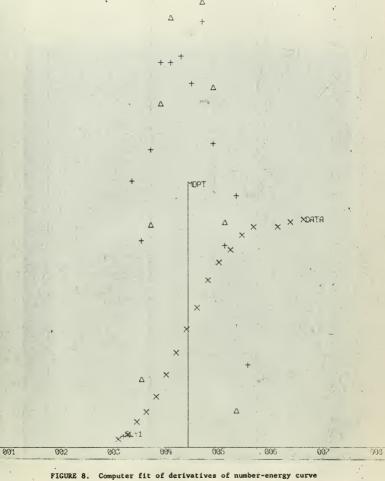


FIGURE 8. Computer fit of derivatives of number-energy curve to a gaussian distribution.



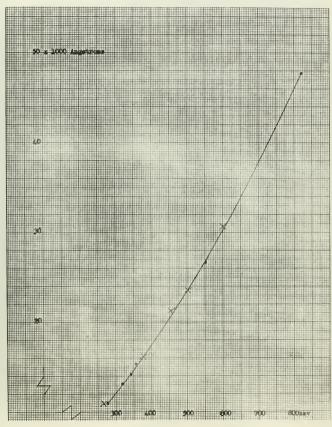


FIGURE 9.



## 5. Acknowledgements.

The author wishes to express his appreciation for the guidance and encowagement received from Associate Professors E. A. Milne and D. E. Harrison, Jr. in this research. The technical assistance of technicians R. A. Garcia, M. J. Andrews, and M. J. Odea in the construction of the target chamber and vacuum system is also greatly appreciated.



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